IS 11720-2 (2005): Methods of Test for Synthetic Rubber, Part 2: Measurement of Vulcanization Characteristics with Oscillating Disc Curemeter [PCD 13: Rubber and Rubber Products]

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IS 11720 (Part 2) : 2013
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भारतीय मानक

सिंथेटिक रबड़ की परीक्षण पद्धतियाँ

भाग 2 दोलन डिस्क क्योरमीटर से वल्कनीकरण लक्षणों का मापन

(दूसरा पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR SYNTHETIC RUBBER

PART 2 MEASUREMENT OF VULCANIZATION CHARACTERISTICS WITH THE OSCILLATING DISC CUREMETER

(Second Revision)

ICS 83.060

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

January 2013 **Price Group 6**

Rubber and Rubber Products Sectional Committee, PCD 13

NATIONAL FOREWORD

This Indian Standard (Part 2) (Second Revision) which is identical with ISO 3417 : 2008 'Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Rubber and Rubber Products Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The first revision of this standard published in 2005 under dual numbering system and this second revision is being carried out to align it with the latest published International Standard on the subject.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

The technical committee has reviewed the provision of the following International Standard referred in this adopted standard and has decided that it is acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 6502 : 1999	Rubber — Guide to the use of curemeters

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR SYNTHETIC RUBBER

PART 2 MEASUREMENT OF VULCANIZATION CHARACTERISTICS WITH THE OSCILLATING DISC CUREMETER

(Second Revision)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

CAUTION — Certain procedures specified in this International Standard may involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This International Standard specifies a method for determining selected vulcanization characteristics of a rubber compound by means of an oscillating disc curemeter. The use of the curemeter is described in ISO 6502.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the reference document (including any amendments) applies.

ISO 6502, *Rubber — Guide to the use of curemeters*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6502 apply.

4 Principle

4.1 A test piece of rubber is contained in a sealed test cavity under an initial positive pressure and maintained at an elevated temperature. A biconical disc is embedded in the test piece and is oscillated through a small rotary amplitude. This action exerts a shear strain on the test piece, and the torque required to oscillate the disc depends on the stiffness (shear modulus) of the rubber. The torque is recorded autographically as a function of time.

Direct proportionality between torque and stiffness cannot be expected under all test conditions because — particularly in higher torque ranges —elastic deformation of the disc shaft and driving device have to be taken into account. Moreover, in cases of small amplitudes of deformation, the strain can be expected to have a considerable elastic component. For routine control purposes, however, corrections are not necessary.

4.2 The stiffness of the rubber test piece increases as vulcanization proceeds. The curve is complete when the recorded torque rises either to an equilibrium value or to a maximum value (see Figure 1). If the torque continues to increase, vulcanization is considered to be complete after a given time. The time required to obtain a vulcanization curve is a function of the test temperature and the characteristics of the rubber compound.

4.3 The following parameters can be measured from the recorded curve of torque as a function of time, i.e. $M = f(t)$ (see Figure 1):

M_L	minimum torque;
M_{HF}	plateau torque;
M_{HR}	maximum torque (reverting curve);
M_H	highest torque value attained in a curve where no plateau or maximum value is reached after a specified time;
t_{sx}	time to incipient cure (scorch time);
$t_c(y)$	time to a given percentage of the highest measured torque;
$t_c'(y)$	time from the minimum torque to a given percentage of full cure;
$\frac{100}{t_c(y) - t_{sx}}$	cure rate index (average slope of curve, calculated as indicated by the formula).

The minimum torque M_L depends on the stiffness and the viscosity at low shear rate of the unvulcanized compound.

The highest torque (M_{HF}, M_{HR} or M_H) is a measure of the stiffness of the vulcanized rubber at the temperature of test.

The time to incipient cure t_{sx} is a measure of processing safety.

The times $t_c(y)$ and $t_c'(y)$ and the corresponding torques give information on the progress of vulcanization. The optimum time is often given by $t_c'(90)$.

5 Apparatus

5.1 Curemeter

The curemeter consists of a biconical disc in a temperature-controlled die cavity. The shaft of the disc is secured in a drive shaft and oscillated through a small rotary amplitude (see Figure 2).

The torque applied to the disc represents the resistance of the rubber test piece to deformation and is recorded autographically to yield a curve of torque versus time.

5.2 Die cavity

5.2.1 The dies shall be manufactured from a non-deforming tool steel having a minimum Rockwell hardness of 50 HRC.

The geometry of the dies is shown in Figures 3 and 4. Suitable means shall be employed, by the design of the dies or otherwise, to apply pressure on the test piece throughout the test in order to minimize slippage between the disc and the rubber. Holes shall be drilled in both the upper and lower dies at the locations shown in Figures 3 and 4 to enable temperature sensors to be inserted. The surfaces of the die cavity shall contain rectangular-shaped grooves located at 20° intervals to minimize slippage. The lower die dimensions shall be as given in Figure 3. The upper die shall contain identically shaped grooves. The dimensions of the upper die shall be as given in Figure 4.

5.2.2 The lower die shall have a hole in the centre to allow the insertion of disc stem. A suitable low constant friction seal shall be fitted in this hole to prevent material from leaking from the die cavity.

5.3 Die closure

The dies shall be closed, and held closed during the test, by a pneumatic cylinder exerting a force of 11,0 kN ± 0,5 kN.

5.4 Disc

The biconical disc shall be fabricated from a non-deforming tool steel having a minimum Rockwell hardness of 50 HRC. The disc is shown in Figure 5, and the critical dimensions are given in Table 1.

5.5 Disc oscillation

The frequency of the rotary oscillation of the disc shall be 1,7 Hz ± 0,1 Hz except for particular purposes when other frequencies in the range 0,05 Hz to 2 Hz may be used. The maximum angular displacement of the disc shall be 1,00° ± 0,02° about its central position (total amplitude 2°) when the die cavity is empty. If a torque is acting on the disc, the resulting decrease in the angle of oscillation with increasing torque shall be a linear function having a slope within the limits of 0,05°/N·m ± 0,002°/N·m.

Suitable devices shall be provided to verify both the initial amplitude of oscillation and the decrease in amplitude with applied torque.

Other amplitudes may be used when specified for particular purposes. With different frequencies or amplitudes, different results will be obtained.

NOTE An initial amplitude of oscillation of 3° can be used in cases where danger of slippage between test piece and die cavity or disc can be excluded (first of all by regular cleaning of the rotor, see 10.2.3). A higher sensitivity in testing may be obtained at this amplitude, which may be useful in production quality control.

5.6 Torque-measuring system

5.6.1 Measurement

A device which produces a signal that is directly proportional to the torque required to turn the disc shall be used to measure the torque acting on the disc.

5.6.2 Recording

A recorder with a paper feed shall be used to record the signal from the torque-measuring device. The recorder shall have a speed of response for full-scale deflection on the torque scale of 1 s or less. The torque shall be recorded with an accuracy of ± 0,5 % of the torque range. Three torque ranges from 0 N·m to 2,5 N·m, 0 N·m to 5 N·m and 0 N·m to 10 N·m shall be provided.

Although the procedure is written for a pen recorder with a paper feed, automatic data acquisition and processing equipment may also be used.

5.7 Temperature measurement

5.7.1 The temperature-measuring systems shall enable the temperature of the dies to be measured to within ± 0,1 °C over the range 100 °C to 200 °C. Calibrated thermocouples, or other suitable temperature sensors, inserted in the dies, shall be used for periodically checking the die temperatures.

5.7.2 The dies shall be mounted in electrically heated aluminium platens. Temperature controllers shall be used to control the temperature of each platen to within ± 0,3 °C at steady state. After insertion of a test piece at 23 °C ± 5 °C, the temperature of the dies shall recover to within 0,3 °C of the test temperature within 3 min.

6 Calibration of torque transducer and recorder

6.1 Provision shall be made for electronic verification of the recorder and torque transducer. One way of doing this is calibration by means of a resistor incorporated in the torque-measuring circuit and which simulates an applied torque of specified value.

6.2 The torque-measuring system shall be calibrated by means of masses or by a standard torque system such as a calibrated torsion spring.

6.3 In order to detect differences between curemeters or changes with use in a single curemeter, tests on reference compounds are useful. The reference compound shall have a shear modulus equal to or greater than the production compounds being tested, and it shall be homogeneous and stable for several weeks. Several tests shall be made on calibrated curemeter(s) in good condition and, from each curve, parameters such as M_H, M_L or t_c' shall be determined. Each set of values obtained for each parameter shall be used to define a confidence interval at a chosen statistical confidence level (95 % or 99 %).

Small changes with use or small differences between curemeters shall not be compensated for if the material parameters measured (M_H, M_L or t_c', for instance) are within the confidence intervals. In such a case, the differences observed are not statistically significant.

The cause of large deviations, i.e. the cause of statistically significant variations detected when one of the parameters is no more within its confidence interval, shall be determined and necessary repairs or maintenance performed.

7 Test piece

A test piece approximately 30 mm in diameter and 12,5 mm in thickness or of the equivalent volume shall be used for each test. Preferably, the test piece should be cut from a previously sheeted sample, which shall be as free from air as practical. A total volume of 8 cm^3 for the test piece is considered optimum.

NOTE Suitable test piece size is assured if a small amount of compound is allowed to extrude between the edges of the dies. Oversized test pieces cool the cavity excessively during the early part of the test cycle and invalidate the test.

8 Curing temperature

The curing temperature is determined by the nature of the rubber compound or the application, but will normally be in the range 100 °C to 200 °C. The tolerances on the curing temperature shall be ± 0,3 °C.

9 Conditioning

The test piece shall be conditioned at 23 °C ± 5 °C for a minimum of 3 h before testing.

10 Procedure

10.1 Preparation for test

Bring the temperature of both dies (see 5.2) to the curing temperature, with the disc (5.4) in place and the die cavity in the closed position. With the disc in place and the dies closed, adjust the recorder pen to the zero-torque line on the chart. Position the pen at the zero-time position on the chart. Calibrate the recorder if needed (see 6.1) and select the correct torque range (see 5.6.2).

10.2 Loading the curemeter

10.2.1 Open the dies, place the test piece on top of the disc, and close the dies within 5 s. When testing sticky compounds, insert some suitable thin-film material below the rotor and above the test piece to keep the compound from sticking to the dies.

10.2.2 Time shall be counted from the instant the dies are closed. The disc may be oscillating (see 5.5) at zero time or started not later than 1 min after the dies are closed. The curve is complete when the recorded torque rises either to an equilibrium value or to a maximum value. If the torque continues to increase, vulcanization is considered to be complete after a given time.

10.2.3 A deposit of material from the rubber compounds under test may build up on the disc and dies. This may affect the final torque values. It is suggested that reference compounds be tested daily to detect this occurrence. If such contamination develops, it may be removed by very light blasting with a mild abrasive. Extreme care shall be used in this operation to retain sharpness of serrations and not change dimensions. Ultrasonic cleaning or cleaning with hot solvents or non-corrosive cleaning solutions may also remove the deposit. If solvent or solution cleaning is used, the first two sets of results after cleaning shall be rejected.

11 Expression of results

11.1 General

The applicable values of those indicated in 11.2 to 11.5 shall be taken from the cure curve.

11.2 Torque values

M_L minimum torque, in newton metres;

M_{HF} plateau torque, in newton metres;

M_{HR} maximum torque (reverting curve), in newton metres;

M_H highest torque value attained, in newton metres, in a curve where no plateau or maximum value is obtained after the specified time.

11.3 Time values

t_{sx} time, in minutes, to an increase of x tenths of a unit of torque above M_L (see 11.4 and 11.5);

$t_c(y)$ cure time, in minutes, to y % of full torque development (see 11.5);

$t_c'(y)$ cure time, in minutes, for torque to increase from the minimum torque M_L to $M_L + 0{,}01y(M_H - M_L)$ (see 11.4).

11.4 Times to different percentages of full cure

Unless otherwise specified, it is recommended that the following specific parameters be used:

t_{s1} time, in minutes, for torque to increase to 0,1 N·m above M_L;

$t_c'(50)$ time, in minutes, for torque to reach $M_L + 0{,}5(M_H - M_L)$;

$t_c'(90)$ time, in minutes, for torque to reach $M_L + 0{,}9(M_H - M_L)$.

If an amplitude of 3° is used instead of the standard 1°, t_{s2} shall be used in place of t_{s1}; i.e. the time, in minutes, for the torque to increase to 0,2 N·m above M_L.

11.5 Cure rate index

$100/[t_c(y) - t_{sx}]$ parameter representative of the average slope of the cure rate curve in the steep region.

12 Test report

The test report shall include the following information:

a) sample details:

 1) a full description of the sample and its origin,

 2) details of the compound tested;

b) test method:

 1) a reference to this International Standard,

 2) details of the curemeter used;

c) test details:

 1) the nominal amplitude of oscillation, reported as half the total displacement, i.e. 1° for a total displacement of 2°,

 2) the frequency of oscillation, in hertz, if other than 1,7 Hz (see 5.5),

 3) the torque range selected, in newton metres,

 4) the paper feed speed of the recorder, in millimetres per minute,

 5) the heating-up time, in minutes (see 5.7.2.),

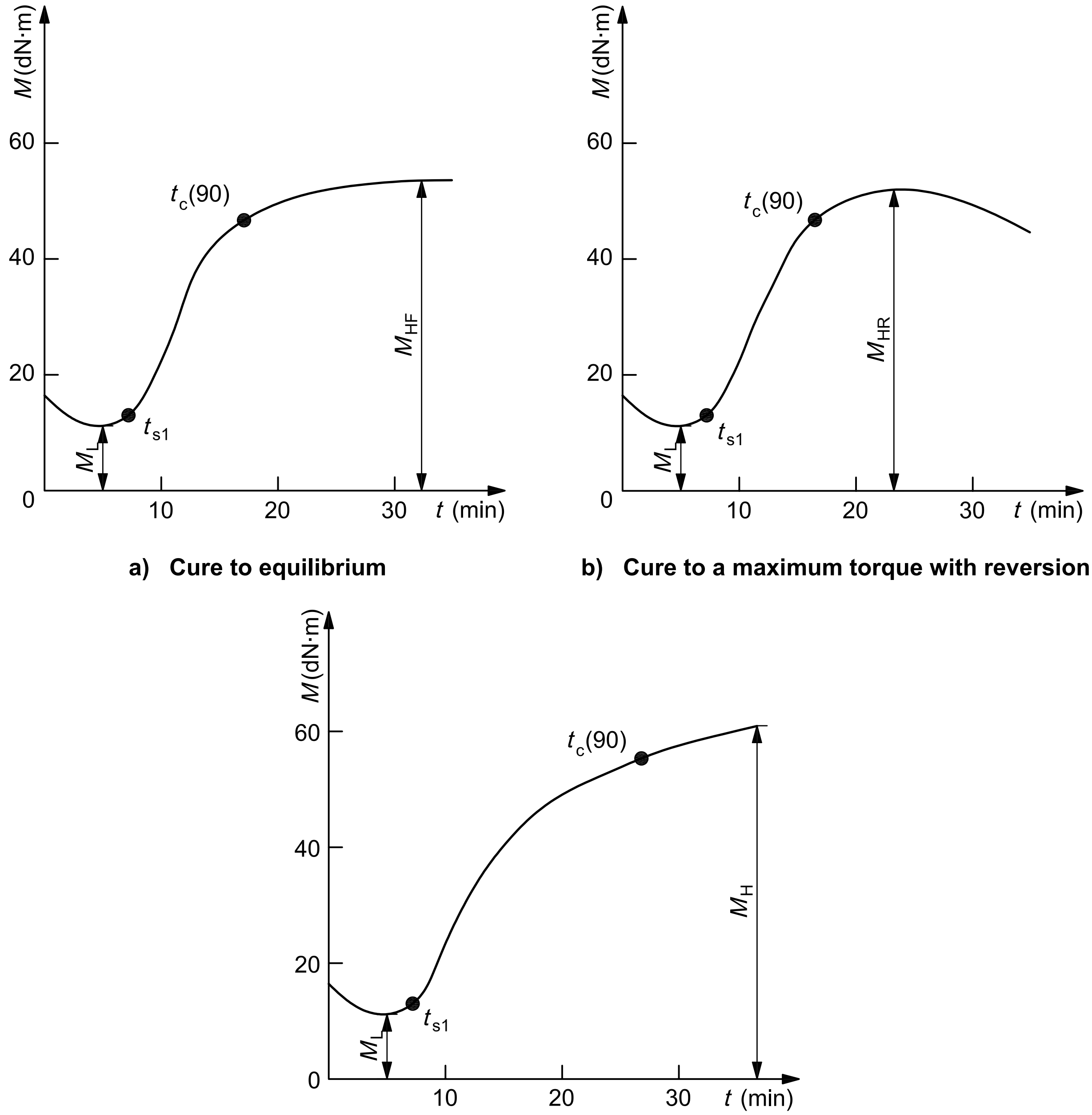
 6) the curing temperature, in degrees Celsius;

d) test results:

 1) the type of vulcanization curve obtained (see Figure 1),

 2) the test results read from the curve;

e) the date of the test.

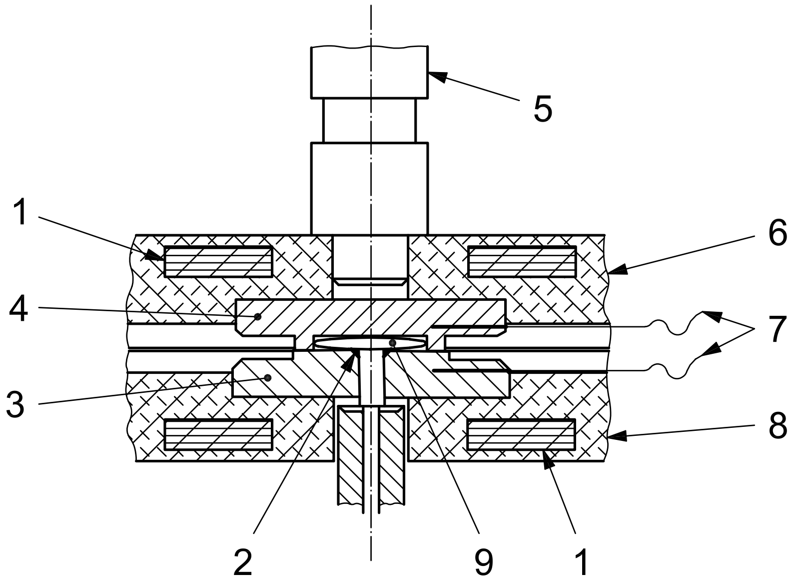


a) Cure to equilibrium

b) Cure to a maximum torque with reversion

c) Cure to no equilibrium in maximum torque

Figure 1 — Types of vulcanization curve



Key

1 heater

2 seal

3 lower die

4 upper die

5 cylinder rod

6 upper platen

7 calibrated temperature sensors

8 lower platen

9 biconical disc

Figure 2 — Curemeter assembly

Dimensions in millimetres

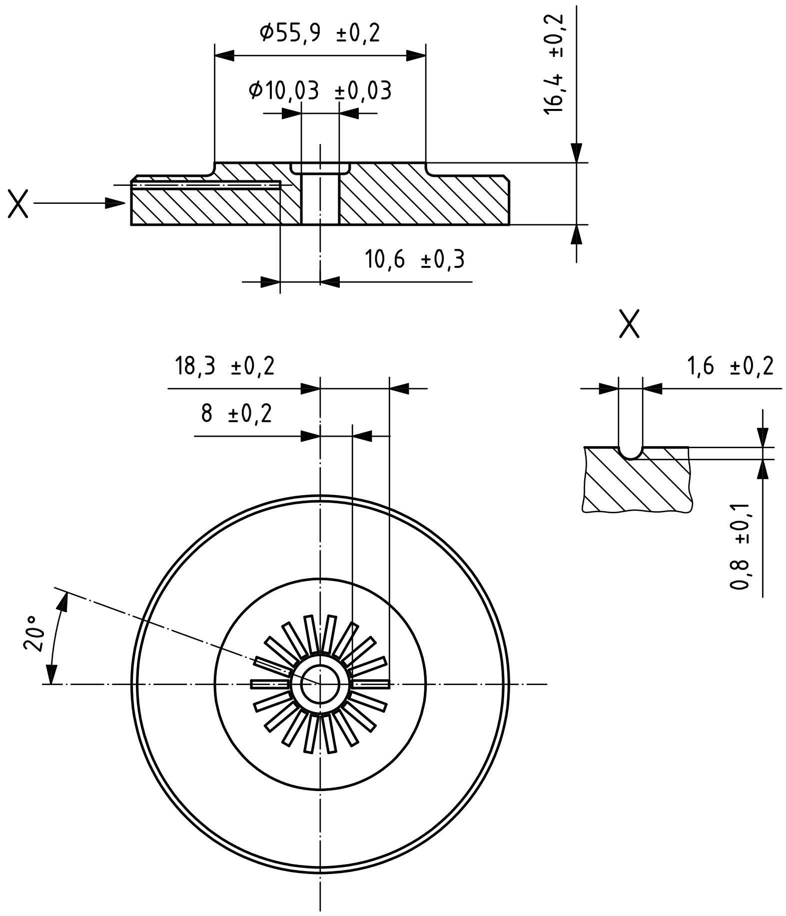


Figure 3 — Lower die

Dimensions in millimetres

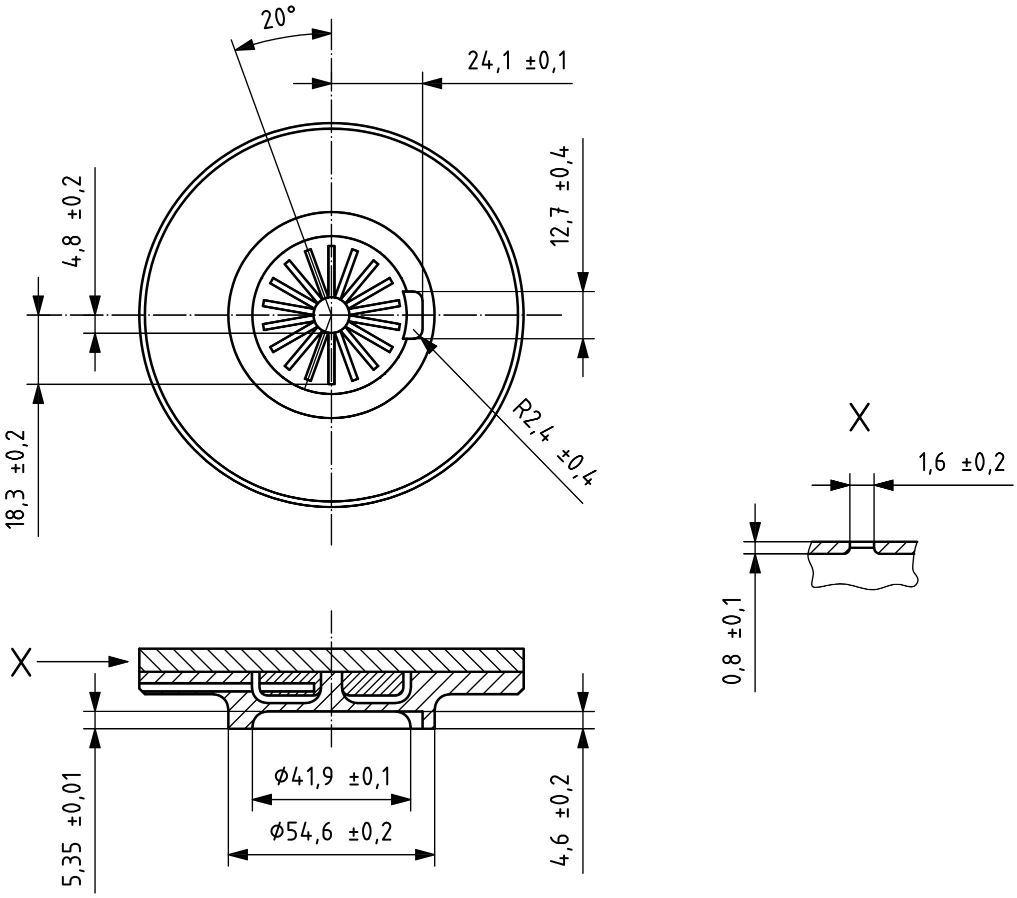
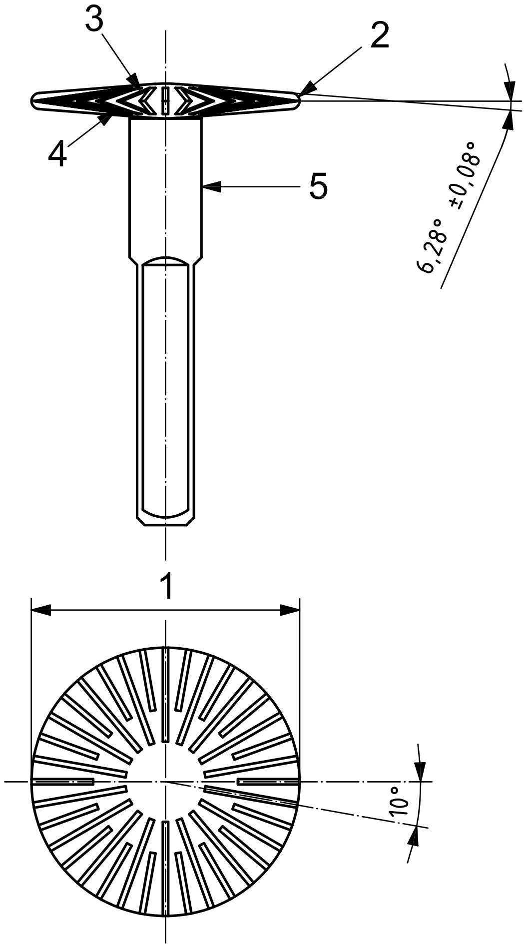


Figure 4 — Upper die



Key

See Table 1.

Figure 5 — Biconical disc

Table 1 — Disc dimensions

Dimensions in millimetres

Ref. No.	Dimension		Tolerance
1	Diameter	35,55	± 0,01
2	Radius	0,80	± 0,03
3 [a]	Groove width	0,80	± 0,05
	Groove depth	0,8	± 0,1
	Groove lengths	7,5 min.	
		12,5 min.	
4	Groove width	0,80	± 0,05
	Groove depth	0,8	± 0,1
	Groove lengths	7,5 min.	
		9,5 min.	
5	Diameter	9,51	± 0,01
	Length of the circular part of the disc shaft	20,0	± 0,5
	Length of the square part of the disc shaft	35,0	± 0,5

[a] The grooves on the top and bottom surfaces shall be staggered by 5°.

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amendment No.	Date of Issue	Text Affected

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